

Eurachem Workshop on 'Uncertainty from Sampling and Analysis for Accredited Laboratories': BAM, Berlin, 19-20 November 2019

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EURACHEM WORKSHOP ON 'UNCERTAINTY FROM SAMPLING AND ANALYSIS FOR ACCREDITED LABORATORIES'

BAM, Berlin, 19-20 November 2019

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Eurachem Workshop on 'Uncertainty from sampling and analysis for accredited laboratories', was held in conjunction with Eurolab-Germany and CITAC, at BAM in Berlin on November 19th-20th 2019.

This two-day Workshop attracted over 140 participants from 27 countries, who made 30 presentation, both orally and as posters. One of its objectives was to launch the Second Edition of the Eurachem/CITAC Guide on Measurement Uncertainty arising from Sampling (UfS) (reviewed on page 60 of this Newsletter). The first day was therefore mainly focused on UfS and several of the new ideas in this area that have been incorporated into the revised Guide. For example, the Uncertainty Factor was explained as a better way to express measurement uncertainty (U) when the values are large (e.g. $U > 20\%$), and when the frequency distribution of the uncertainty is shown to be log-normal, rather than the Gaussian that is usually assumed. Some examples were given where this asymmetry in the uncertainty was seen to arise from the sampling process, but other examples arose from purely analytical sources, such as the determination of genetically modified organisms (GMOs) in soya.

Another general objective of the Workshop was to understand how UfS relates to the accreditation of sampling, that has an increased role in the latest version of ISO/IEC 17025, explained in contributions from ILAC and UKAS. Another interesting presentation reviewed over 100 published applications of UfS estimation, since the publication of the First Edition of the UfS Guide in 2007. These ranged across many application sectors, mainly environmental (70%, of which 40% on water, and 30% on soil and sediment), but also on food safety (22%) and industrial processes (8%). This review set the

scene for discussion of the way forward for research and applications in UfS. Suggestions for new areas of application included estimating uncertainty of *in situ* measurements (e.g. sensors), where the sample is not removed but left undisturbed and therefore unmixed. The uncertainty of *in situ* measurements needs, therefore, to include the UfS caused by the heterogeneity of the analyte in most test materials in nature. Also recognized was the need to find better ways of allowing for high values of UfS in compliance assessment. The first review and meta-analysis of UfS estimates across a whole application sectors, has been published for food (Ellison *et al.*, 2017, *Analytical Methods*, 9, 5989-5996). Two potential benefits of applying this type of approach to other applications sectors were identified. The results may help to substantiate the general applicability of the mathematical model between UfS and analyte concentration that was found across the food sector. Secondly, such models may allow regulators to predict approximate values of UfS that can be incorporated into new regulatory guidelines.

On the second day, the earlier discussion of the uncertainty factor for UfS on particular, lead on to discussions of the more general topic of expressing high levels of uncertainty and its asymmetric distribution, regardless of its origins. There were some differences between what presenters considered the limits to be for various levels of uncertainty. Low levels of uncertainty were generally considered to be when the relative standard uncertainty (u') is below either 10 or 15% (i.e. expanded U' below 20 or 30%), with high levels classified as being over either $u' = 40\%$ or 50% , and a medium level in between these two limits. There was also a range of

views on how uncertainty should be expressed at each level. Most speaker suggested that at low levels of uncertainty, uncertainty should be expressed as relative expanded uncertainty, unless the analyte concentration was close to the detection limit (e.g. $s < 2 \times$ limit of quantitation), when the expanded uncertainty should be used. For medium and high levels of uncertainty, use of the expanded uncertainty factor (FU) was generally recommended. Although FU is usually calculated by taking logarithms to the base 'e' of the measured analyte concentration, the same values are obtained if logarithms taken to the base 10. It was recognized that the microbiological sector has been expressing uncertainty using logarithms taken to the base 10, but not expressed as FU .

There was increasing appreciation of Monte Carlo (MC) simulation in studies of uncertainty. In one presentation MC simulation was used to show that at low uncertainty levels ($u' < 15\%$) there was no significant discrepancy between normal, log-normal and MC distributions. Above this level ($u' > 15\%$), the prediction from the normal distribution progressively diverged from those of both the log-normal and the Monte Carlo simulation, which were virtually identical. The conclusion was that the distribution of the values attributable to the measurand was lognormal rather than normal.

One innovative presentation described how to calculate the confidence interval of an uncertainty estimate. The equations used to make these calculations for classical ANOVA applied to a normal distributed data have been published earlier, but not previously incorporated into packages for uncertainty estimation. The innovation here was to extend this procedure to deal with data sets with up to 10% of outlying values, requiring the application of robust ANOVA. The boot-strapping approach was applied to calculate the 95% confidence interval of the estimates of uncertainty arising from both the chemical analysis and the sampling. These intervals can then be used to compare estimated of uncertainty made by difference approaches, to test whether the apparent differences are statistically significant.

The case for using more empirical information from quality control schemes to improve the reliability of

uncertainty estimates, was made by several speakers. There was also discussion the best software both for the estimation of uncertainty, and for research into improving such methods. For the estimation of U and UFS various packages in Microsoft Excel were described (e.g. RANOVA2, and @Risk for MC), but for research purposes, the more flexible packages in the language 'R' were often recommended.

The substantial number of participants and presentations at this Workshop indicate that research into uncertainty estimation, and its applications, are topics of sustained interest and relevance. The many discussions within the Workshop raised numerous new and emerging issues that will ensure that further research into uncertainty and UFS estimation will continue to develop over the coming years. The abstracts and slides from all of the presentations made in the Workshop are available at: <https://www.eurachem.org/index.php/events/workshops/277-wks-mu2019#plenaryDay1>

A proportion of the participants at the Uncertainty Workshop in Berlin in November 2019, are shown below.

